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ELECTRICAL RESISTIVITY OF COMPOSITE SUPERCONDUCTORS

by

Jack H. Davis

and

Jonathan A. Lee

Prepared by

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

Submitted by

The University of Alabama in Huntsville
School of Mathematical and Natural Sciences
Huntsville, Alabama 35899

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I. INTRODUCTION

In addition to its superconducting properties, a superconductor is usually characterized by poor thermal conductivity[10] and relatively high electrical resistivity in the normal state. To remedy this situation in this paper, we present a study of superconducting properties of Cu-rich CU-Nb wires prepared by directionally solidified and cold-rolled technique. Some of the specimens were prepared by melting, directional solidification and diffusing in Tin. The superconducting tests were conducted at The University of Alabama in Huntsville by J. Davis and student, J. Lee.

A total of 12 wire specimens was tested. The list of the specimens and their compositions is shown in Table 1. The first 6 specimens were prepared by and received from Mr. Jim Burka (NASA lab for material processing in Huntsville). The last 6 specimens were received from Mr. E. W. Collings, from Battelle's Columbus Laboratory in Ohio.

In this paper, each specimen from Table 1 was analyzed by plotting experimental data into the following curves:

1) The graph of the residual resistivity as a function of the specimen current at 4.3 K.

2) The graph of the electrical resistivity as a function of the temperature at a constant current.

TABLE 1. CROSS SECTIONAL AREAS OF THE SPECIMENS AND COMPOSITION ARE LISTED.

SAMPLE NO.	CROSS SECTIONAL AREA (cm^2)	ALLOY AND CONDITION
CNA1700	0.0353	Cu-5% Nb As rolled
NbBr169	0.0370	Cu-19.5% Nb-8.5% Sn. Powder Met. Alloy from Battelle, Extruded and Cold Rolled.
81NbBr53	0.043	Same as Specimen NbBr169, Except Directionally Solidified
81CNA51	0.044	Cu-5% Nb-0.25% Al. Directionally Solidified
81CNA47-S	0.011	Cu-5% Nb-0.25% Al. Directionally Solidified, Colded Rolled and Tin Diffused. (2.4% Sn)
81CNA47-B	0.047	Cu-5% Nb-0.25% Al. Directionally Solidified and Tin Diffused (3.0% Sn)
A-3	1.6×10^{-3}	Cu-Nb Alloy
A-4	2.54×10^{-4}	Cu-Nb Alloy
A-5	3.84×10^{-5}	Cu-Nb Alloy
B-1	0.012	Cu-Nb Alloy
B-4	5.83×10^{-4}	Cu-Nb Alloy
C-5	0.0353	Copper 99.9%

FIGURE 1 Sample holder and its components:
1. Current Probe
2. Voltage Probe
3. Sample
4. Resistance Thermometer

II. EXPERIMENTAL PROCEDURE

The 4 probe technique of electrical resistivity measurement was used to detect the specimen superconductivity. A specimen holder and its components are shown in Figure 1. The specimen holder is a small piece of IC board (4 x 2.5 cm). On this board there are four gold coated, elastic clip probes which hold the specimen securely. The 2 outer probes pass a known current through the specimen, and the voltage developed across the inner probes is then measured by using a nanovoltmeter. The distance between the inner probes is about 1.6 cm. The voltage is proportional to the electrical resistance of the specimen. The resistivity is found by knowing the dimensions of the specimen. For references purposes, the gross cross sectional areas of the specimens are listed in Table 1.

In order to measure the temperature, a resistance thermometer is also attached to the specimen holder. There are two resistance thermometers being used: the Platium-Resistance-Thermometer (PRT) and the Germanium-Resistance-Thermometer (GRT). The PRT is used when the temperature is between 60 K to 300 K. Below 60 K, the GRT is used since the PRT loses its sensitivity to use at low temperature.

In addition, a SPDT control switch is used to reverse the direction of the applied current through the specimen.

III. RESULTS AND DISCUSSION

A. Resistivity-Current Relations at 4.3 K

Figure 2 shows the current-voltage relations at 4.3 K for sample C-5, 81NbBr53, 81CNA51, 81CNA1700. Sample C-5 represents copper (99.5% pure) which has a linear current-voltage relation. This results in a constant value of resistivity as a function of specimen current showing in Figure 2A. The resistivity value for copper is slightly fluctuated around 70 nano ohm-cm, which is acceptible for reference purpose[1]. Above the C-5 curve is the current-voltage relation for specimen 81NbBr 53. This specimen produces a non-linear I-V relation which has a sharp decreasing slope from 0.5 to 2.5 amp current range. From 2.5 to 5.5 amps, the I-V slope is constant. This results in a linear resistivity-current relation as shown in Figure 2B. Below 0.8 amp, the R-I curve seems to exhibit some negative resistivity values in small magnitudes. Sample 81CNA51 has almost the identical I-V curve of sample 81NbBr53; however, 81CNA51's curve has slightly higher value in resistance. The results in resistivity-current relations are shown in Figure 2C. Note that the sample tends to have small negative resistivity values when the sample current is below 0.25 amp. Sample 81CNA1700 shows a very interesting current-voltage relation. From 0.5 to 7.5 amps, the voltage response is very small but when the current is greater than 7.5 amps, the voltage response increases rapidly. This unique I-V relations resembles the common-base output current-voltage characteristic of a p-n-p transistor[2]. The resistivity-current relation for sample 81CNA1700 is shown in Figure 2D. Note the sharp rise in resistivity around 7.5 amps. For all 12 tested specimens, only 3 specimens did not show negative resistivity: Specimen C-5, and 81CNA1700, and

81CNA47-S. Figure 3 shows the current-voltage relations for sample A-3, A-4, B-1, and NBNR169. Note that for current ranges from 0 to 8.5 amps, the specimen's voltage response is small in magnitude (about 3.5 micro-volt maximum). Samples A-3, A-4, and B-1 show very interesting I-V characteristics that is when the positive current is applied the sample produced a negative voltage response. This results in the negative resistivity-current characteristics shown in Figures 3a and 3b; however, these negative values of resistivity are small in magnitude (in pico-ohm cm). Sample NbBr169 shows that for a small value of positive applied current the response voltages are negative. Above 0.13 amps, the voltage is positive generating the resistivity-current characteristics as shown in Figure 3c.

Figure 4 shows current-voltage relations for samples A-5 and B-4. For small value of positive-applied current, the voltage response is negative. This results in negative resistivity-current characteristics shown in Figures 4a and 4b for A-5 and B-4, respectively. For sample A-5, the positive resistivity exists when the current is greater than 4.8 amp. For sample B-4, the positive resistivity requires current greater than 2.6 amp.

Figure 5 shows current-voltage relations for samples 81CNA47-B and 81CNA47-S. A small negative voltage response exists for 81CNA47-B but this effect could be observed in detail by using the plot of resistivity versus current as shown in Figure 5a. However, for sample 81CNA47-S, negative resistivity did not appear even with a small positive applied current.

From the R-I relations, we may conclude that most of the tested specimens exhibit critical currents 8.5 A at 4.3 K. Sample 81CNA1700 shows a sharply defined value of critical current; however, its normal resistivity value has not been recorded. Presumably a higher current is

required for A-3, A-4 and B-1 would show its normal resistance. Some specimens (A-3, A-4, B-1) did not exhibit critical current at 4.3 K. Note that if this is the case, the critical-current densities for A-3, A-4, and B-1 should be very high since these specimens have small cross sectional areas. Specimen 81CNA47-S has a small but measurable resistivity at 4.3 K, even with very small value of applied current. However, this specimen also has the normal-resistivity characteristics when applied current of 1 amp is reached.

When a positive current is applied to the specimen, some of the specimens produced a negative voltage response. Not all tested specimens shown this negative effect. The 3 specimens that did not shown negative resistivity are 81CNA1700, 81CNA47-S and C-5 (being 99.9% pure Cu). The negative voltage response for a positive applied current is most pronounced for specimens A-3, A-4, and B-1. Up to 8.5 amp maximum applied current, the response voltage is still recorded as negative. Unfortunately, the negative voltage response is small in magnitude but also measurable as seen in Appendix I.

Battelle's lab's specimens exhibit negative resistivity over a considerable current range. For a higher value of applied current (with respect to the current range), some of these Battelle's specimens will cross the zero-resistivity axis and exhibit positive resistivity. It was thought that NASA's specimens did not give this negative effect. Thus the R-I relations for NASA's specimens did not show the negative effect on the previous progress reports[3]. However, when all NASA specimens were tested again using smaller applied currents, these specimens started to show negative resistivity effect. Thus for NASA specimens the negative effect exists for only small current range.

As mentioned earlier, when a positive current is applied is some of the specimens, it produced a negative voltage response. This negative effect has not been noticed in literature. This negative resistivity might be related to several possibilities; the superconductive tunnelling effect[9,4] or Josephson's tunnelling effect[10,7] of the normal electron tunnelling effect, since all tested specimens are composite superconductors, e.g., filaments of niobium wires are grown in copper matrices. (Tunnelling effects may result from a system of two superconductors with a thin insulator[11,6]. A less esoteric but more likely cause might be thermal emf., heating effects by the applied current, or reliability of the experiment apparatus.

The ohmic heating produced sample thermal profile is invariant[8] with respect to current direction and, thus, is readily subtracted on current reversal. On the other hand, this "negative resistance" is possible due to Peltier junction heating[7,9]. These resulting thermal gradients would also change direction upon current reversal and, thus, would become a non-subtractable-error (Seebeck voltage) in the bulk resistance. Although Peltier heating increases linearly with current, it is apparently overwhelmed by the greater rate at which the current drives the sample normal through the superconducting transition. [Equation (1)]. Typically, a pure element sample Peltier heating is only a few tenths of a microwatt, thus generating a Seebeck voltage of only a few tenths of a nano-volt. However, upon alloying the cryogenic Seebeck coefficients S_A easily increase by one or two orders of magnitude. The current density (I/A) would increase locally in the superconducting filament region. The alloying could decrease the thermal conductivity (K) by one or two orders of magnitude (neglecting the K of the He medium) in Equation (1).

SAMPLE NO.	NEGATIVE RESISTANCE(?)	CRITICAL CURRENT (A/cm^2)
81CNA1700	No	210
NbBr169	Yes	3.50
81NbBr51	Yes	6.37
81NbBr53	Yes	19
81CNA47-S	No	None
81CNA47-B	Yes	4.70
A-3	Yes	$> 4.4 \times 10^3$
A-4	Yes	$> 2.8 \times 10^3$
A-5	Yes	125,000
B-1	Yes	> 560
B-4	Yes	4460
C-5	No	None

TABLE 2 Listed are some of the resulting resistivity-current characteristics of 12 specimens. Note that not all tested specimens have negative resistivity effect; in addition, not all specimens shown critical current for $I < 8.5$ A. Critical current here is define as the current at which the resistivity of the specimen jumps from zero to some positive measurable value.

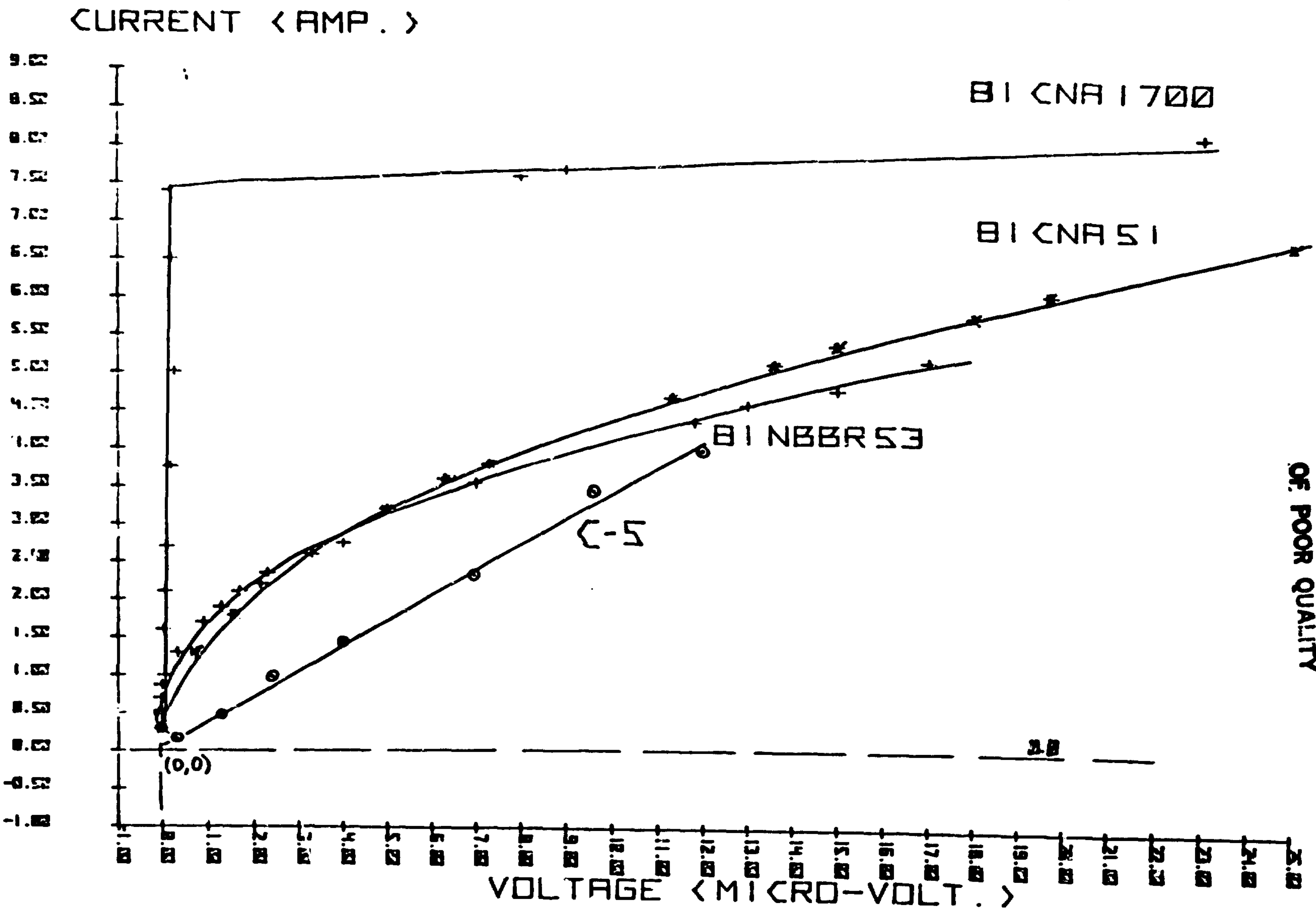

FIGURE 2 Current-voltage characteristics of 4 testing specimens. Note that except for specimen C-5, and 81CNA1700 the rest of the specimens shown all have small values of negative voltage. (T= 4.3 k)

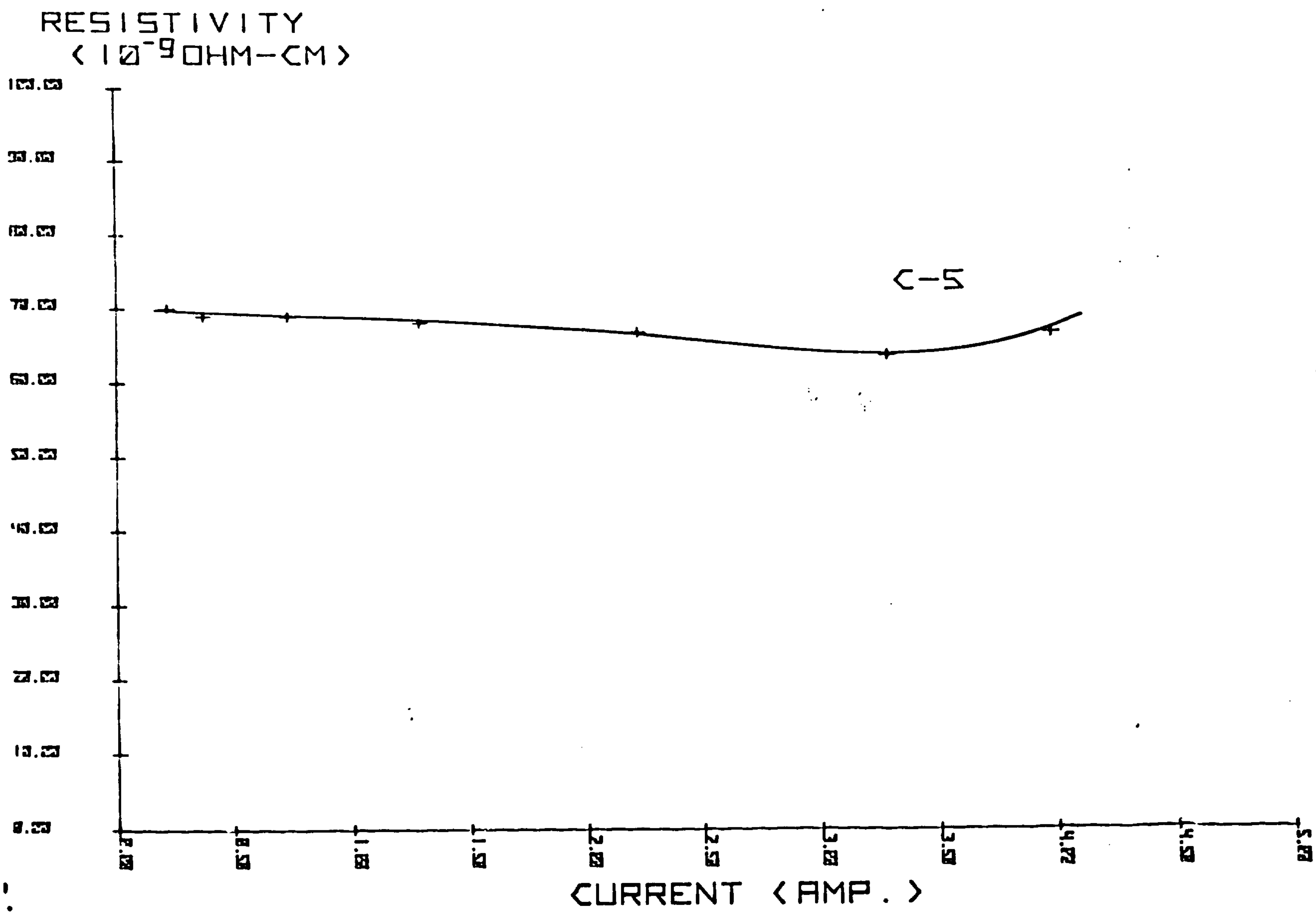

FIGURE 2a. Resistivity-Current characteristics for sample C-5 (99.5% pure copper). (T= 4.3k)

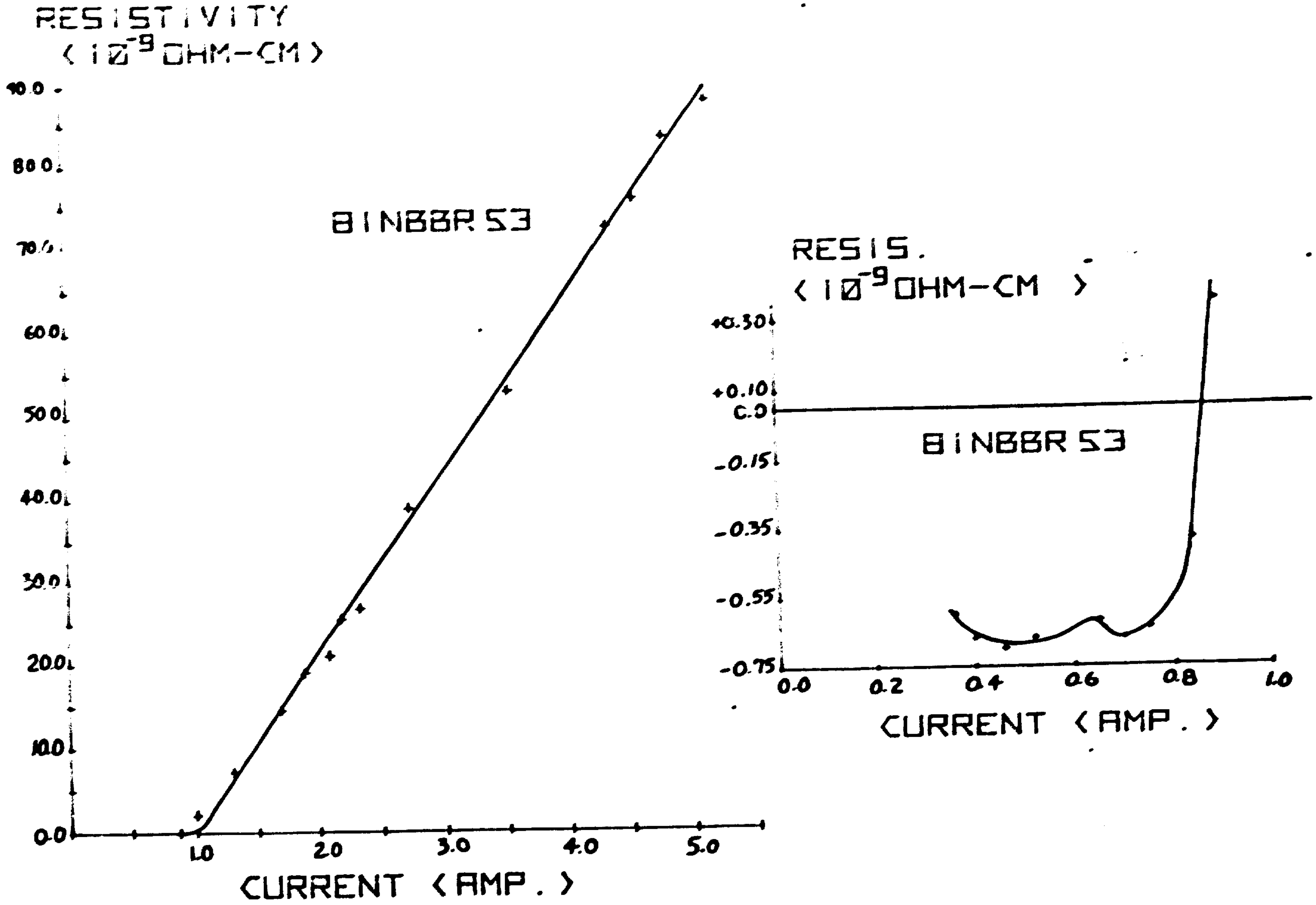

FIGURE 2b. Resistivity-Current characteristics of specimen 81HBBR53. Note that when the applied current is greater than 0.8 amp. the sample shows positive resistance. (4.3 k)

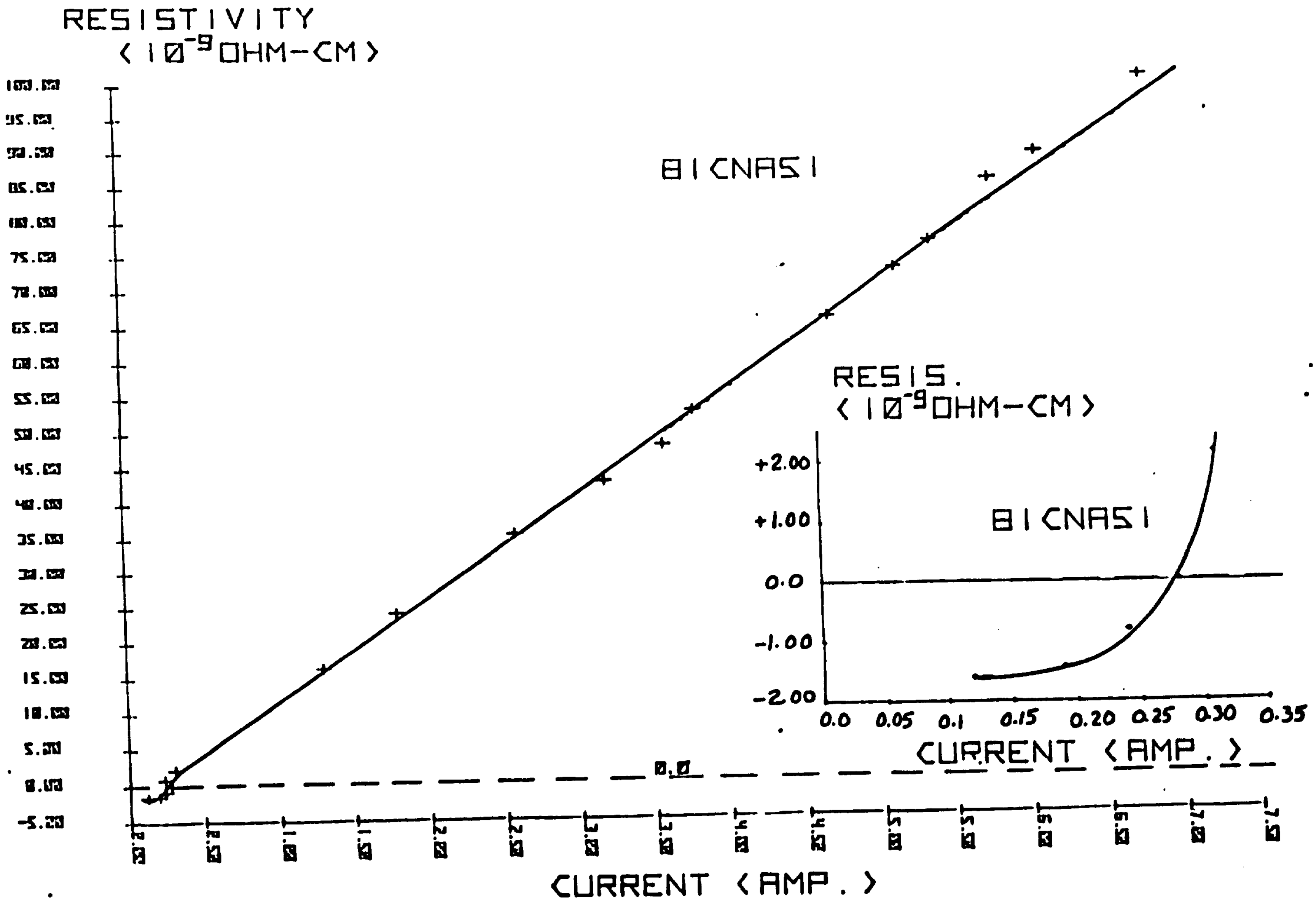

FIGURE 2c. Resistivity-Current characteristics of specimen 81CNA51. Note that when applied current is greater than 0.25 amp. the specimen shows positive resistivity. ($T = 4.3$ K)

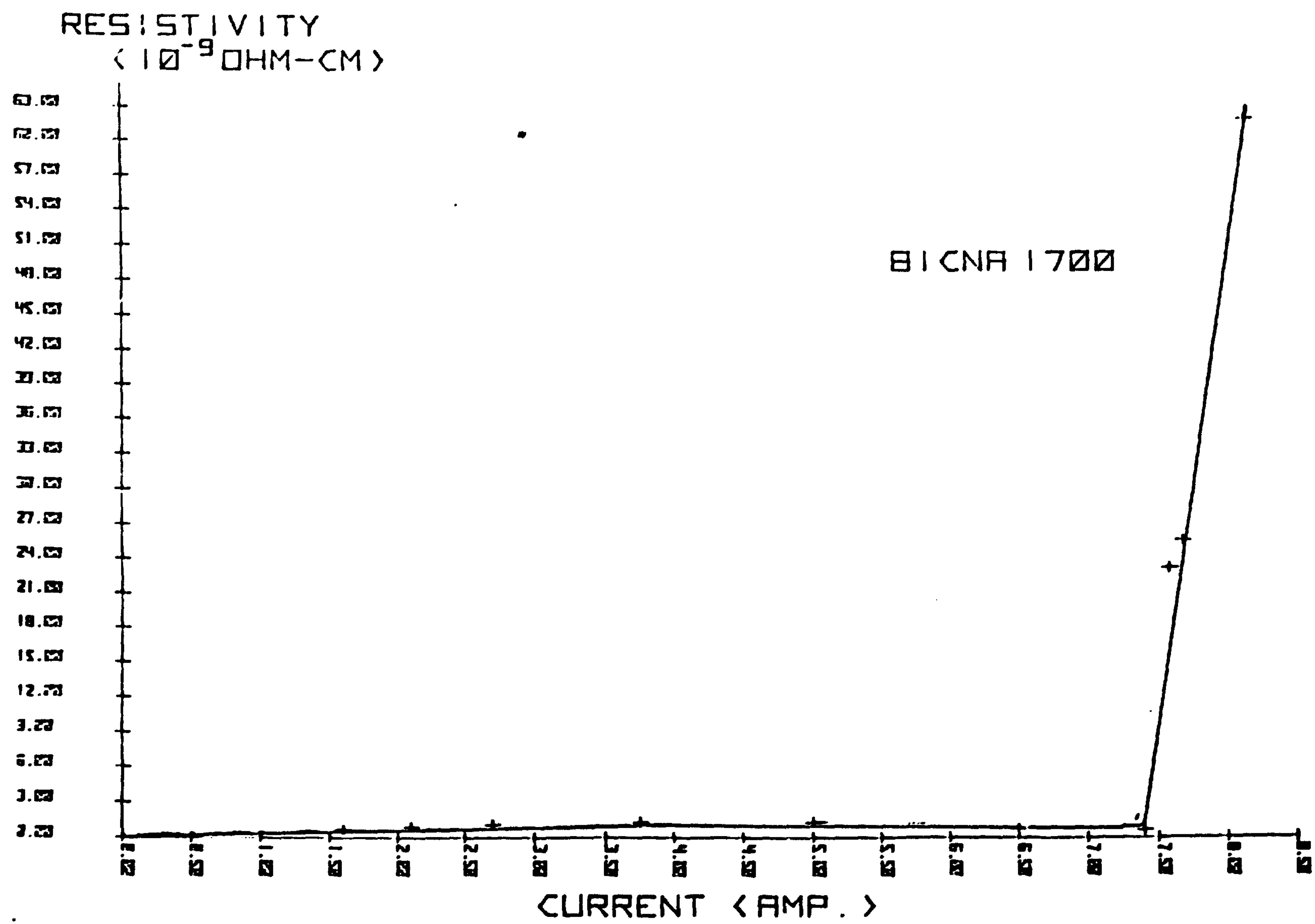

FIGURE 2d Resistivity-Current characteristics for specimen 81CHA1700. Note that there is a constant but small value of resistivity (positive) when the applied current is less than 7.4 amp. ($T = 4.3 k$)

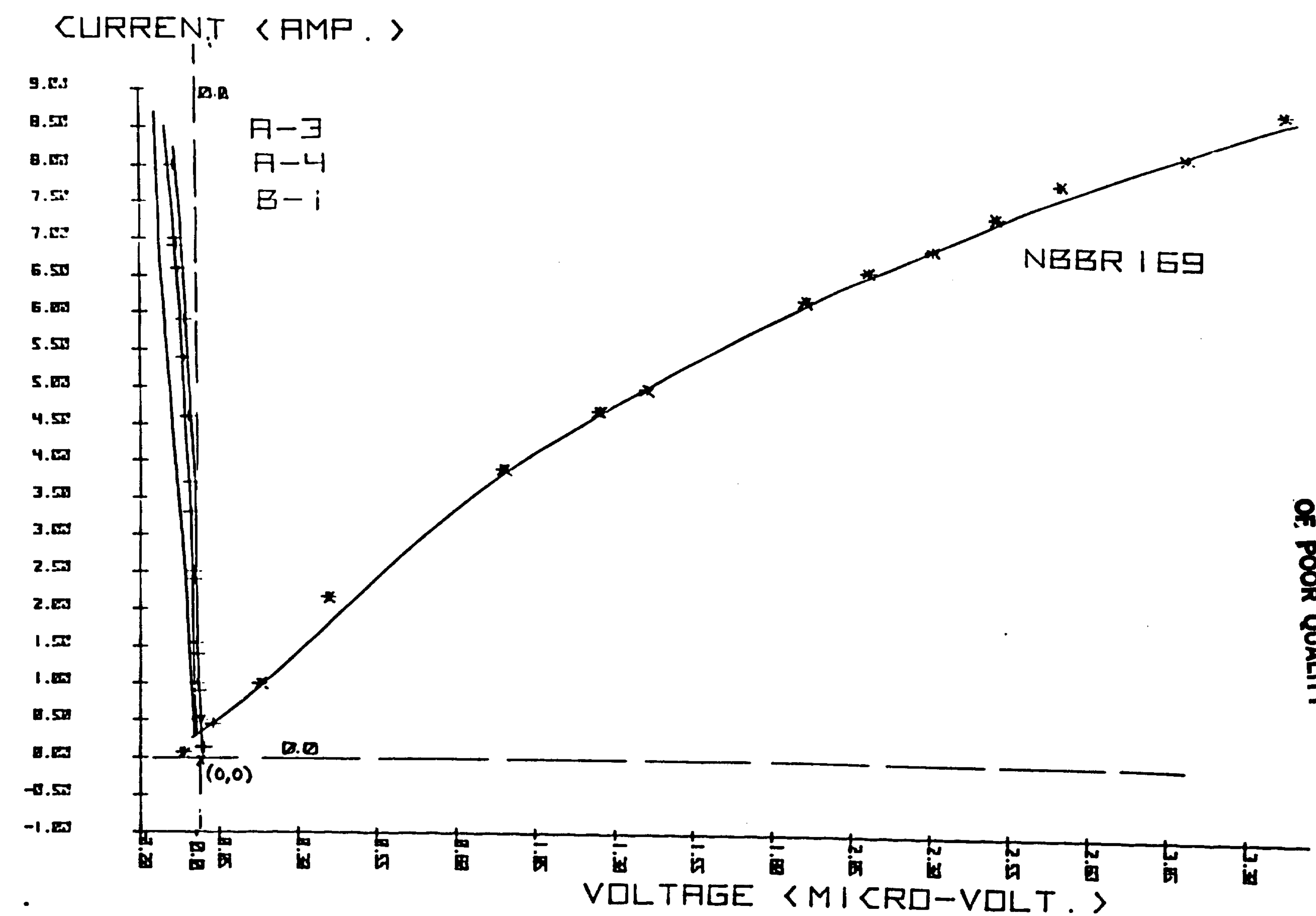

FIGURE 3. Current-voltage characteristics for specimen A-3, A-4, B-1, and NBBR 169. *AT T= 4.3K*

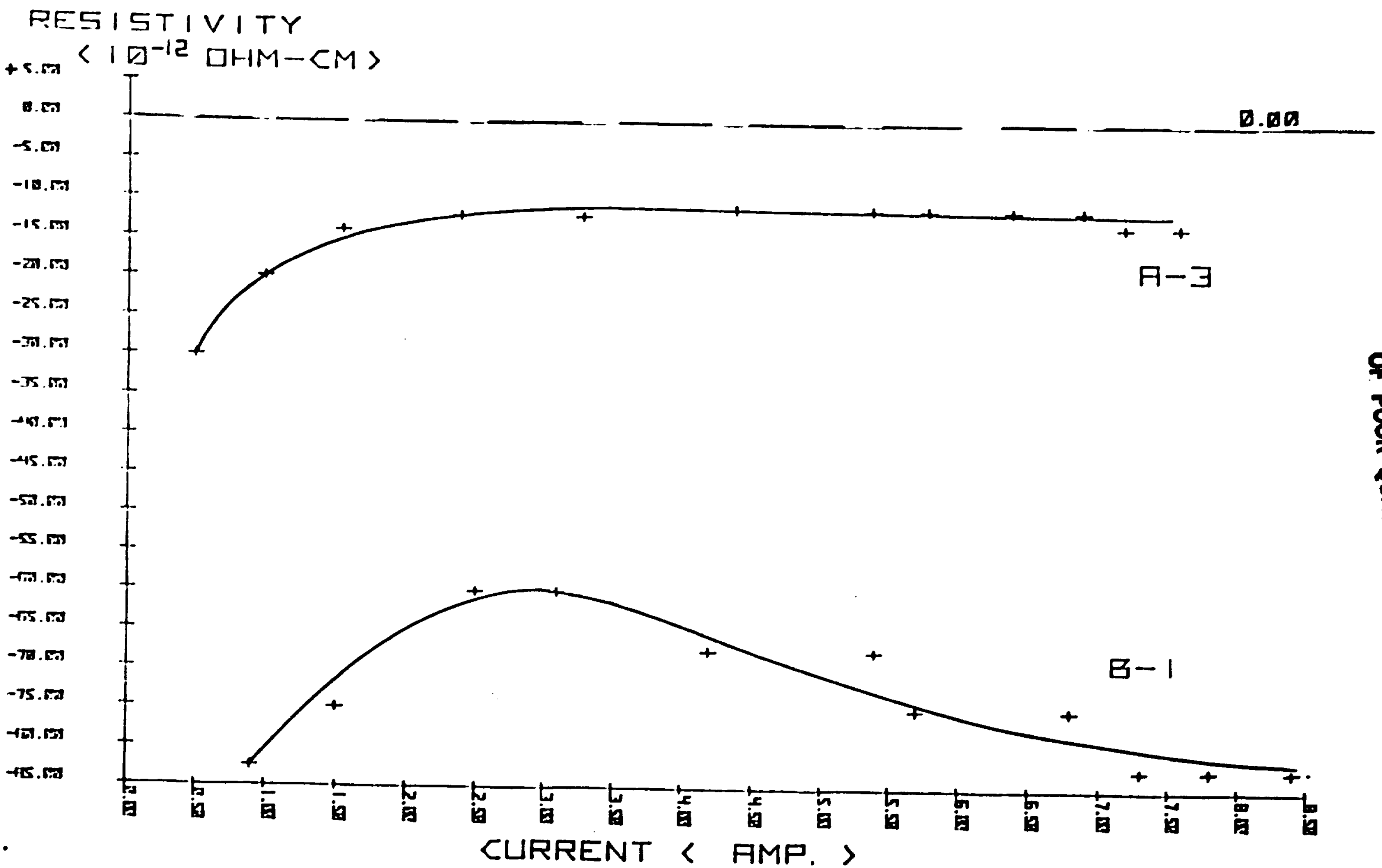

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FIGURE 3a. Resistivity-current characteristics of specimen A-3 and B-1. Note that these specimens have negative resistivity for current range from 0 to 8.5 amp. ($T = 4.3K$)

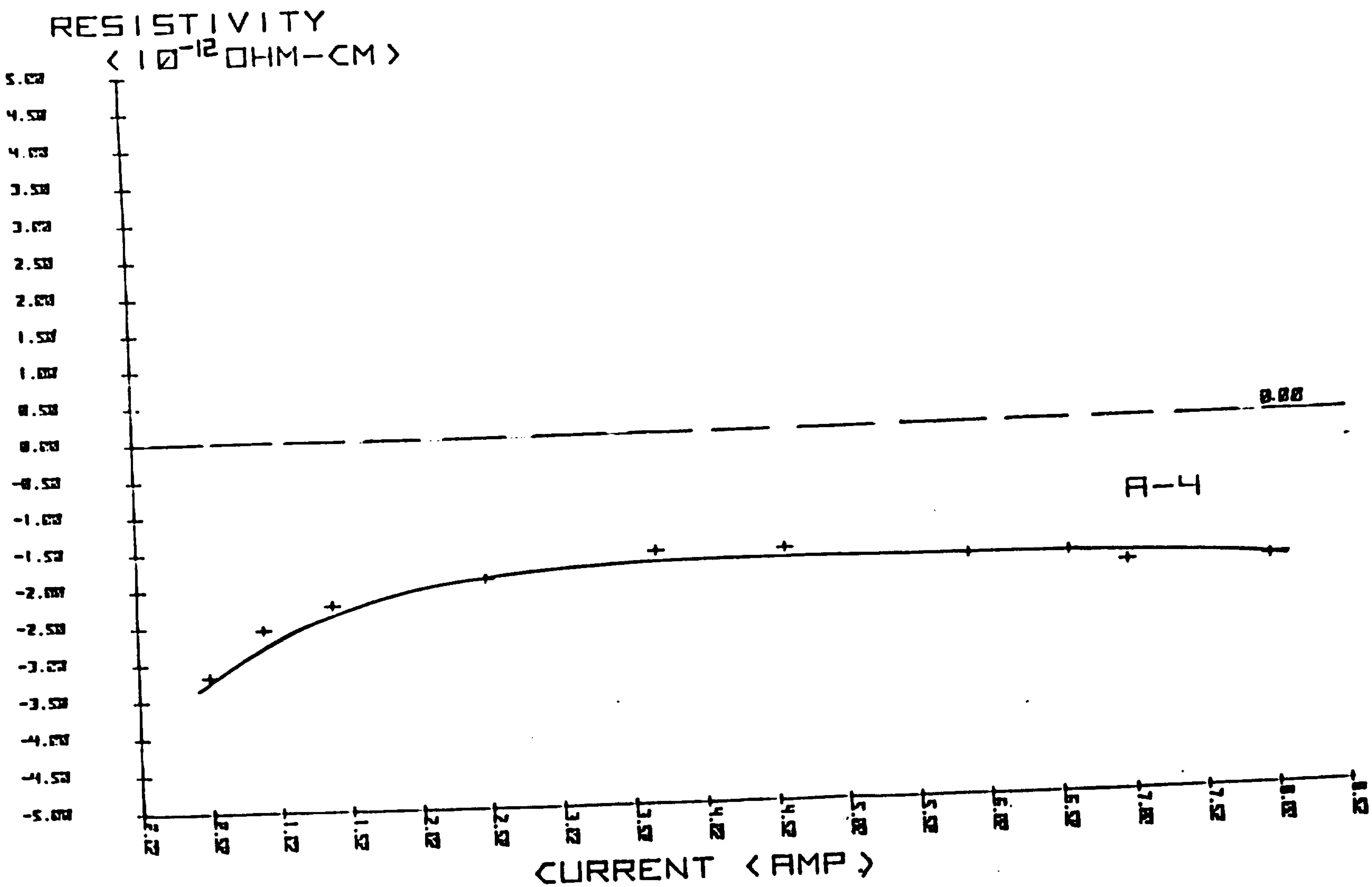

FIGURE 3b. Resistivity- current characteristics for specimen A-4. For current ranges from 0 to 8.0 amps specimen has negative resistivity. T= 4.3k

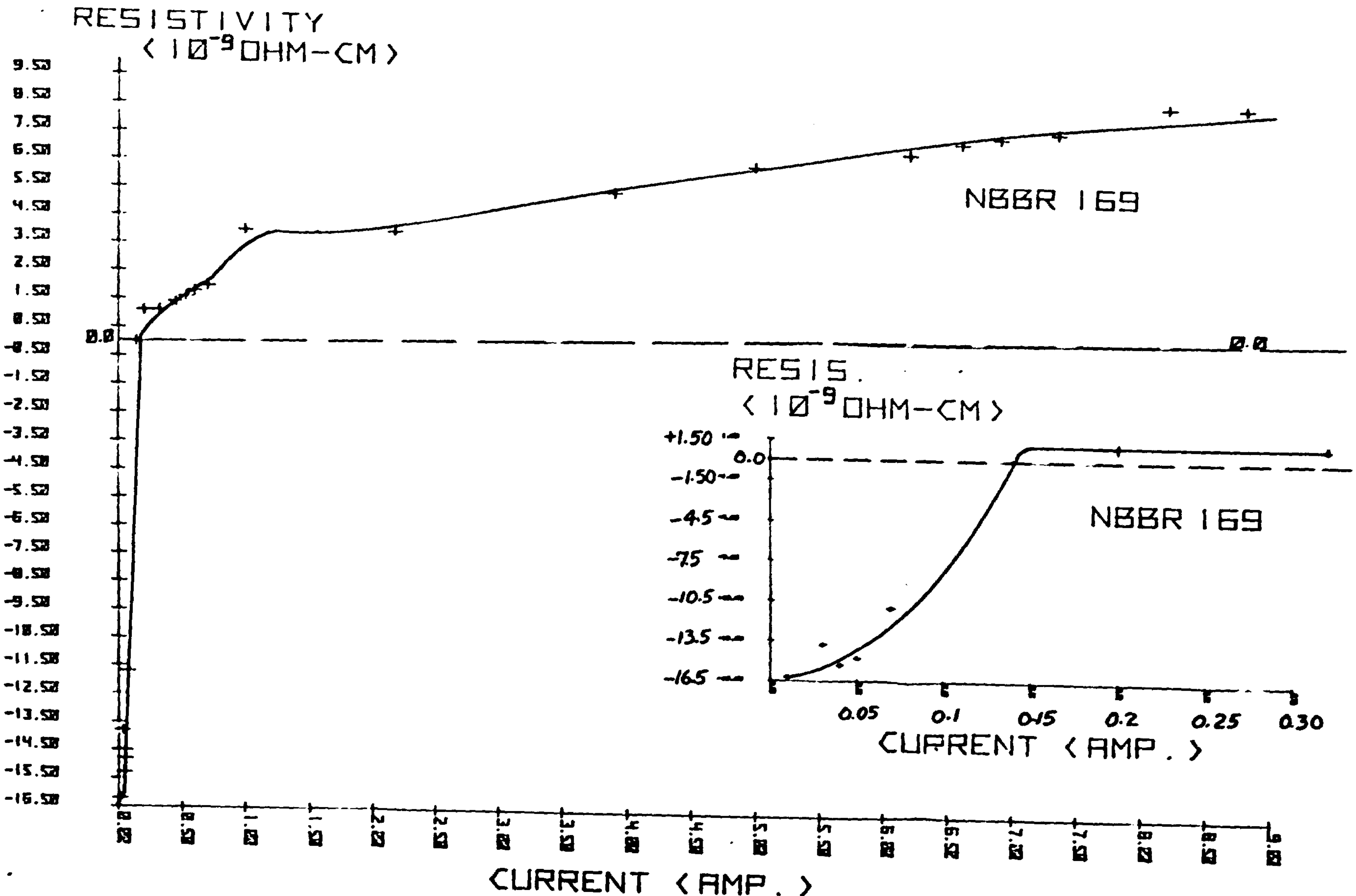

FIGURE 3 c. Resistivity-current characteristics for specimen NBBR 169 at T= 4.3 K.

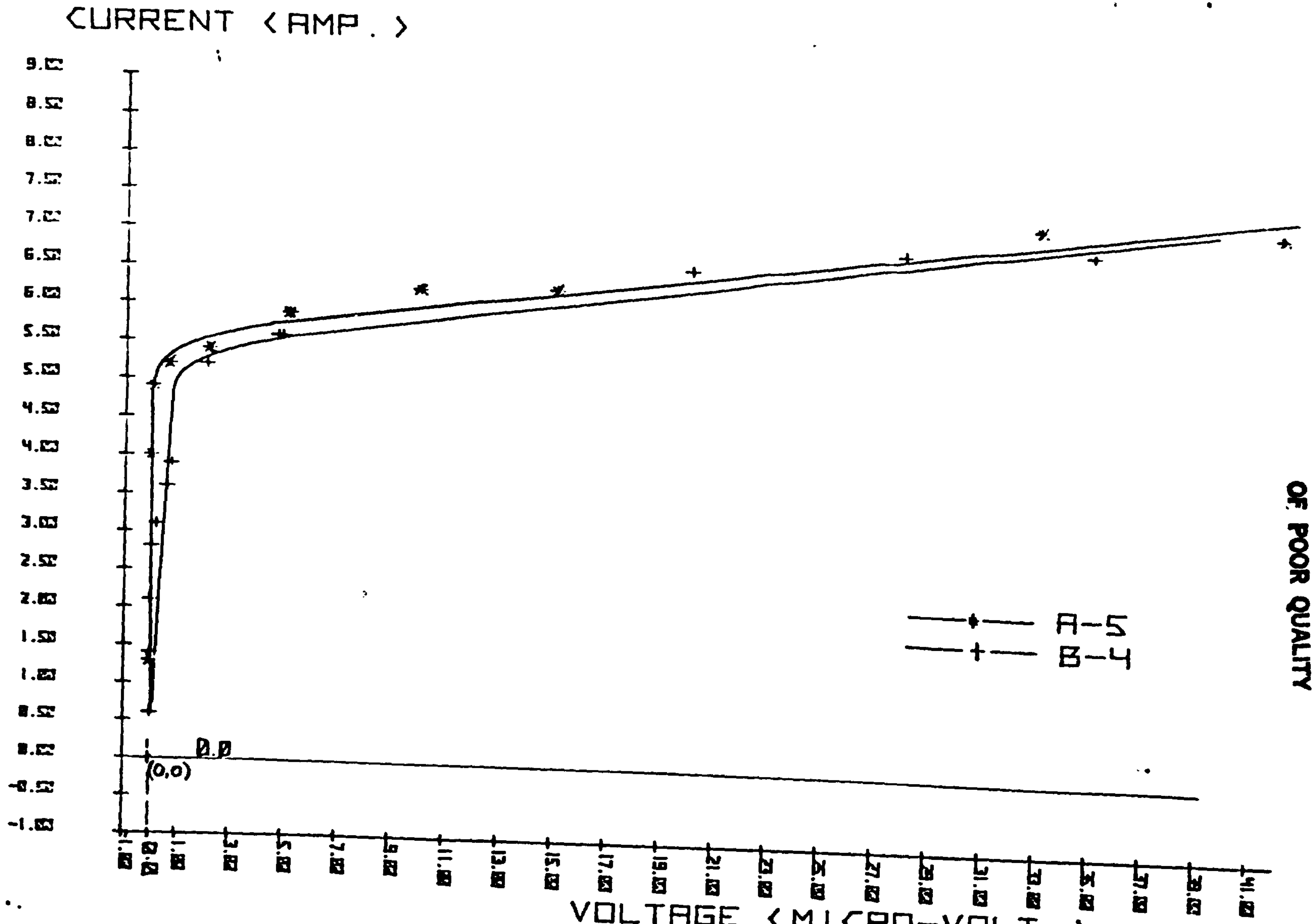

FIGURE 4. Current-voltage characteristics of specimens A-5, B-4. $T = 4.3$ K

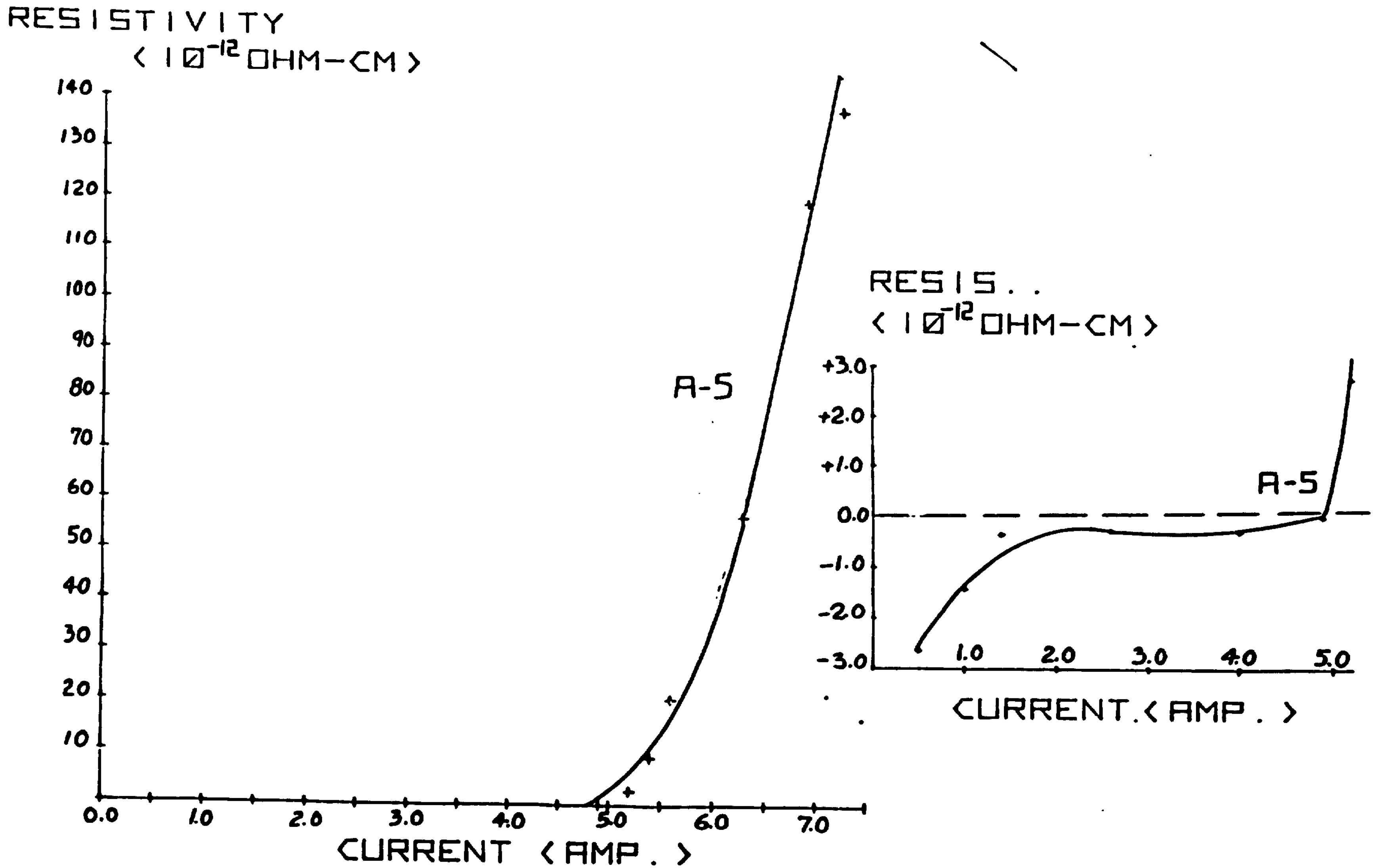

FIGURE 4.a Resistivity-current characteristics for A-5 at 4.3 K

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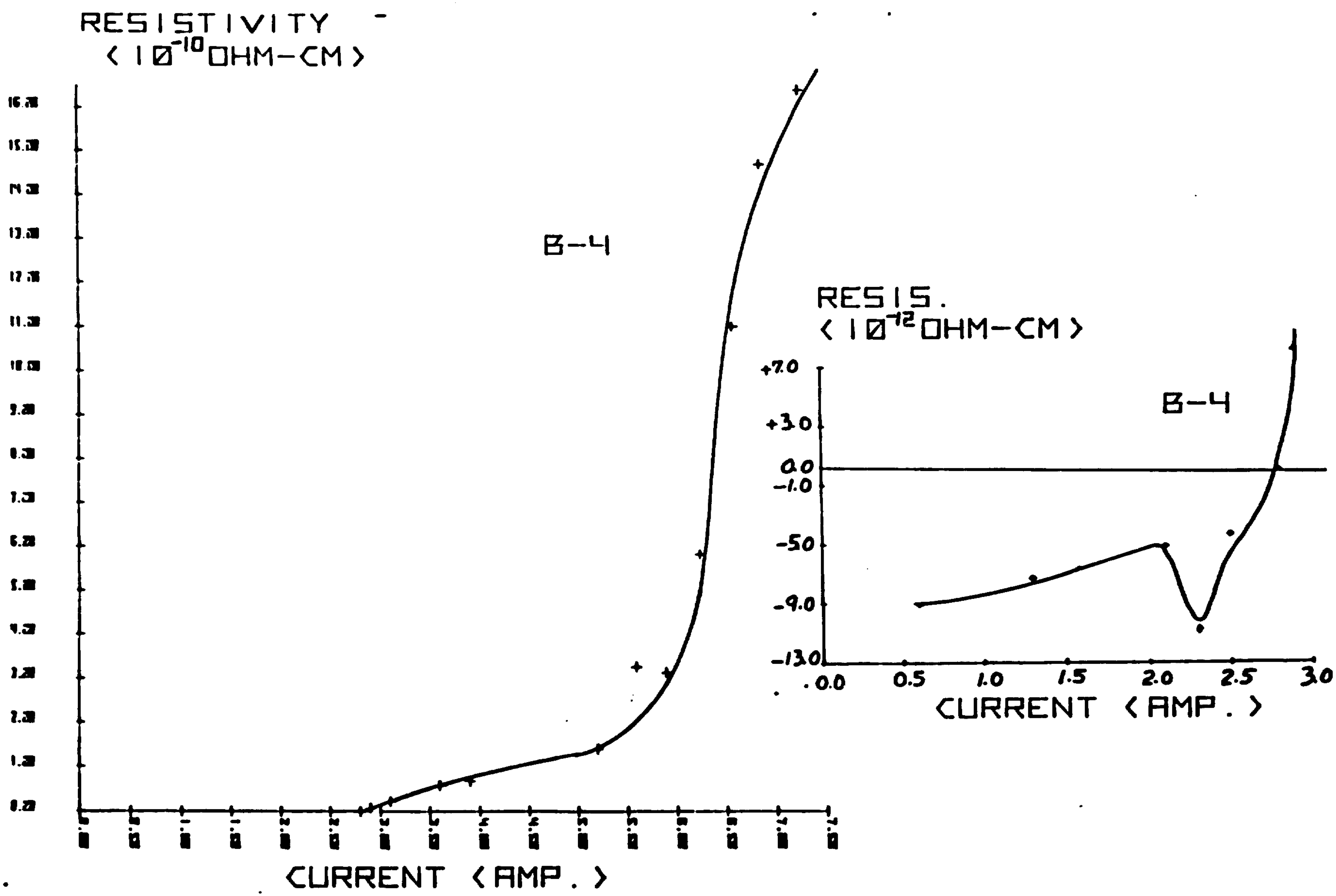


FIGURE 4. b Resistivity-current characteristics of specimenB-4 at 4.3 K

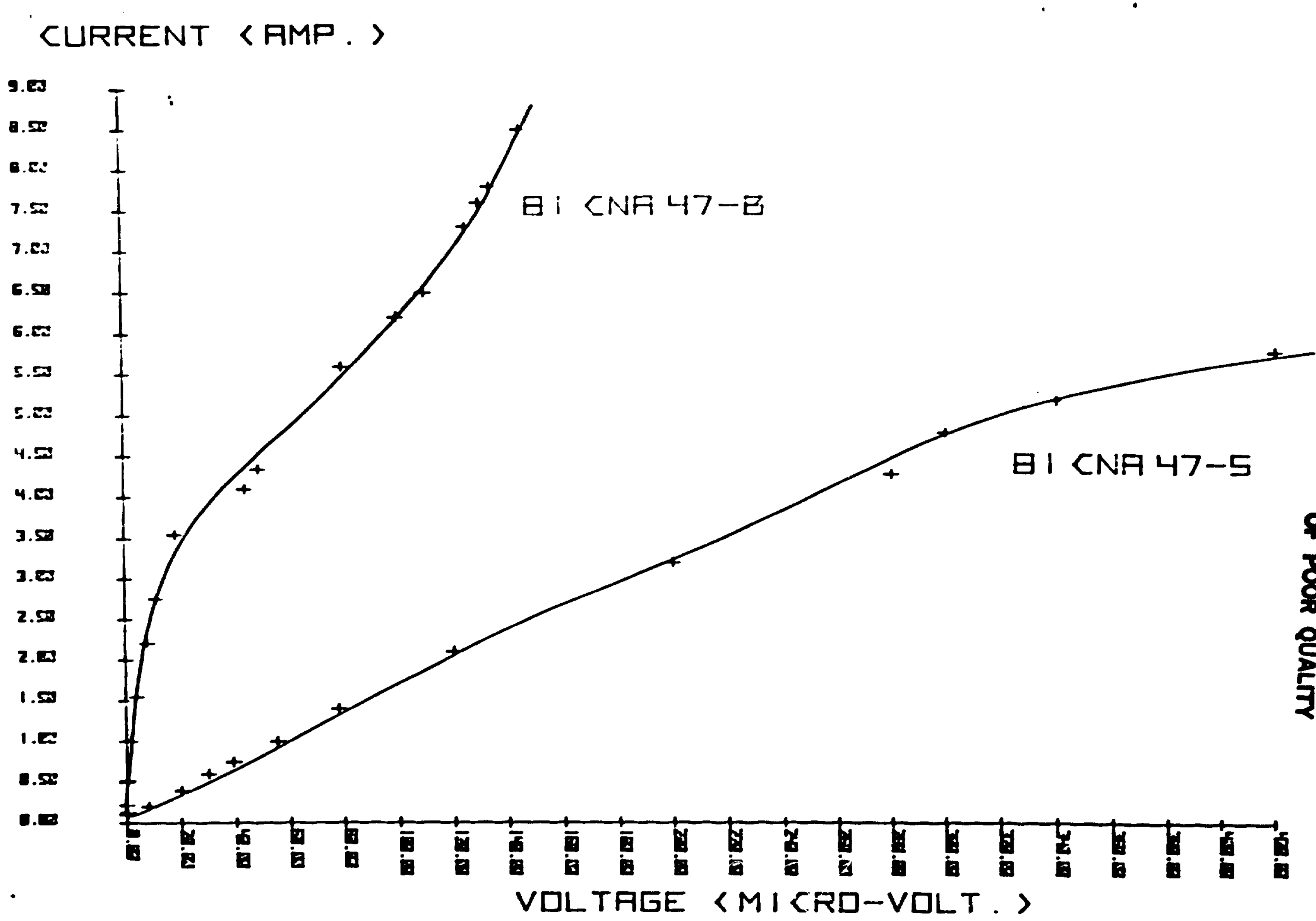


FIGURE 5. Current-voltage characteristics of specimens 81CNA47-B and 81CNA47-S at 4.3 K

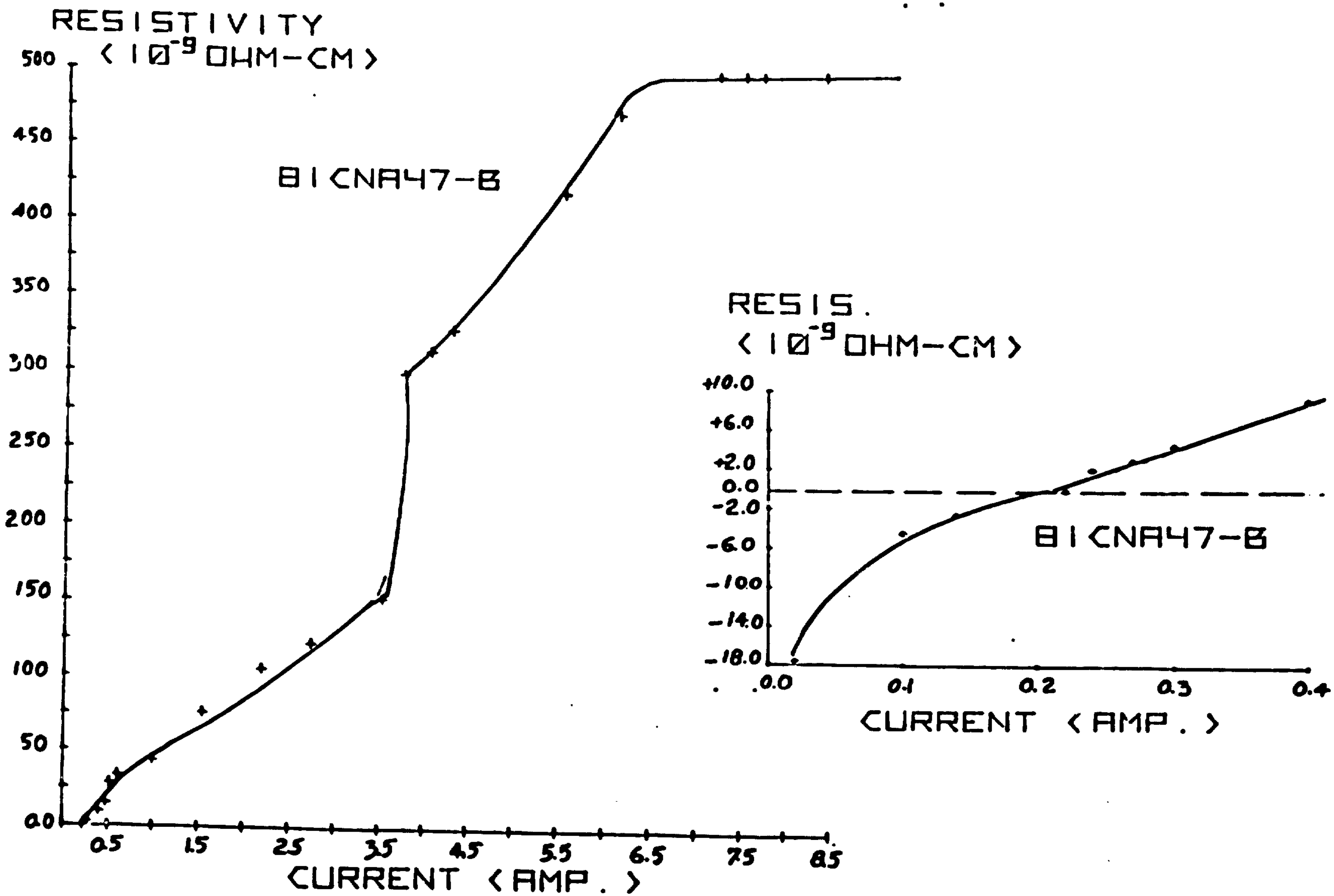


FIGURE 5a Resistivity-Current relations of specimen 81CNA47-B at 4.3 K

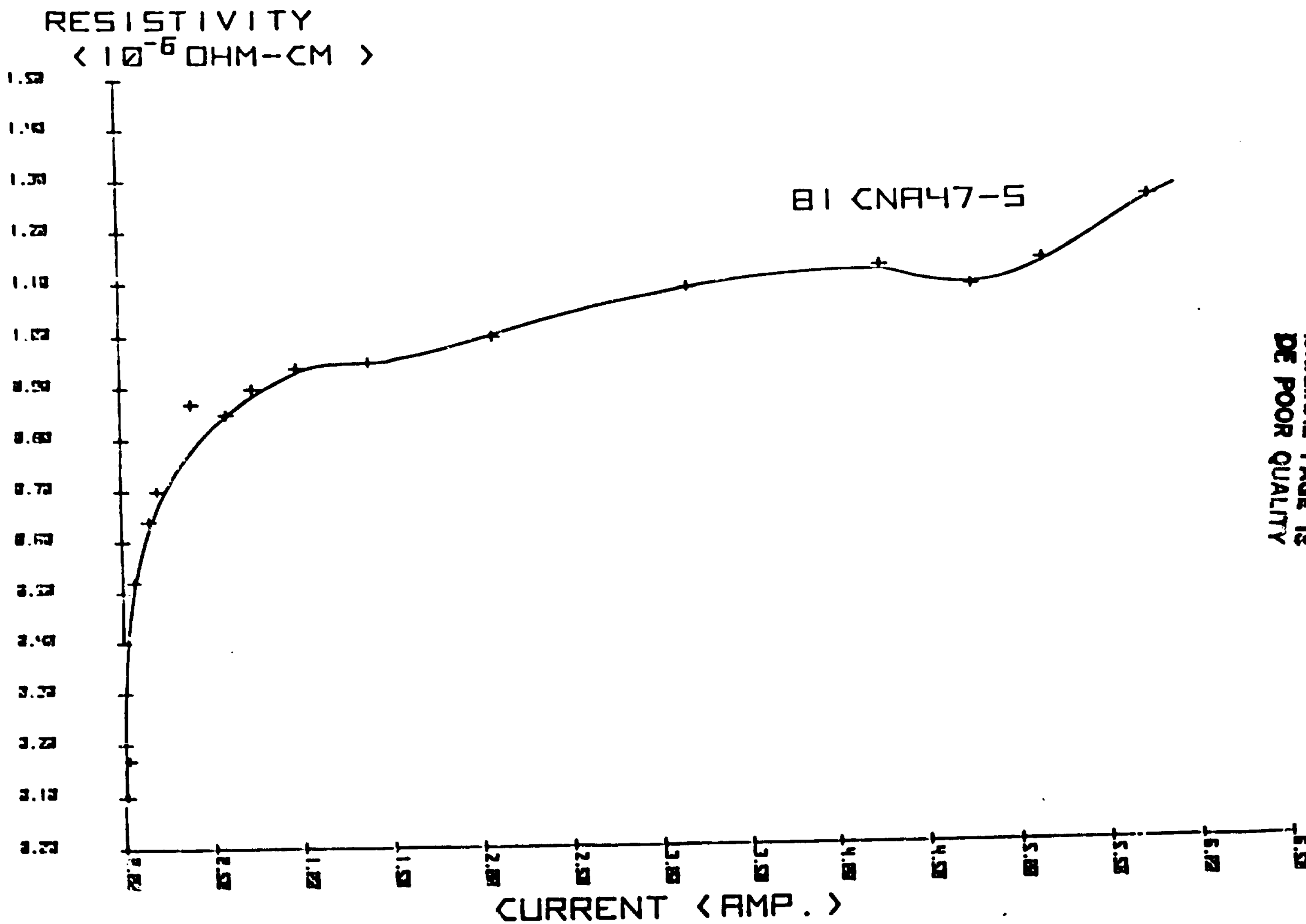


FIGURE 5b. Resistivity-current relations of sample 81CNA47-S at 4.3 K

B. Resistivity-Temperature Relations

Figure 6 shows the electrical resistivity as a function of temperature for 4 NASA specimens in addition to specimen C-5 for equipment calibration purpose. Specimen C-5 (99.5% pure Cu) shows that the resistivity varies from 0.65 to 0.72 micro ohm-cm when the temperature increases from 4.3 K to 14 K. Specimen that has the largest transition temperature range (ΔT) is 81CNA47-B; while NbBr 169 has the smallest transition temperature range, (the smaller the transition temperature range, the higher the slope of the transition curve).

Figure 7 shows the electrical resistivity as a function of temperature for specimen 81CNA47-S which has the largest transition temperature range (ΔT). Also notice that at 4.3 K, the specimen does not give a zero-resistivity value, i.e., a magnitude of 0.5 micro ohm-cm is recorded.

Figure 8 shows that specimen 81NbBr53 has large transition range (from 5.5 K to 14.5 K), of temperature.

Figure 9 shows the electrical resistivity as a function of temperature for 5 specimens from Battelle lab. Specimen A-4 has the highest normal resistivity value when the specimen is at 9.4 K. All specimens shown in this figure have the temperature which ranges from 7.3 K to 9.7 K.

Table 3 is a list of the resistivity-temperature characteristics of all 12 tested specimens. The table shows the specimen's current density used, the normal resistivity, and the temperature range at which the transition of resistivity value takes place as a function of temperature.

$$V = (S_B - S_A)^2 \frac{TIL}{KA} \qquad (1)$$

TABLE 3. A LIST OF RESISTIVITY-TEMPERATURE CHARACTERISTICS FOR 12 TESTED SPECIMENS.

SAMPLE NO.	CURRENT DENSITY (A/cm^2)	TRANSITION TEMP. RANGE (K)	NORMAL RESISTIVITY (Ohm-cm)
NbBr169	7	5.5 to 9.5	0.45×10^{-6}
81CNA1700	5.3	8.5 to 9.3	0.14×10^{-6}
81NbBr51	11.4	4.3 to 8.5	0.40×10^{-6}
81NbBr53	2.3	5.5 to 14.5	8.0×10^{-6}
81CNA47-S	8.8	4.3 to 8.0	1.1×10^{-6}
81CNA47-B	5.3	4.3 to 9.5	0.43×10^{-6}
A-3	62.5	8.0 to 9.4	0.6×10^{-7}
A-4	400	8.2 to 9.3	0.86×10^{-7}
A-5	2604	7.2 to 9.8	0.72×10^{-7}
B-1	8.3	8.5 to 9.2	0.29×10^{-7}
B-4	175	8.0 to 9.0	0.41×10^{-7}
C-5 (99.9% Cu)	14.2	NONE	NONE

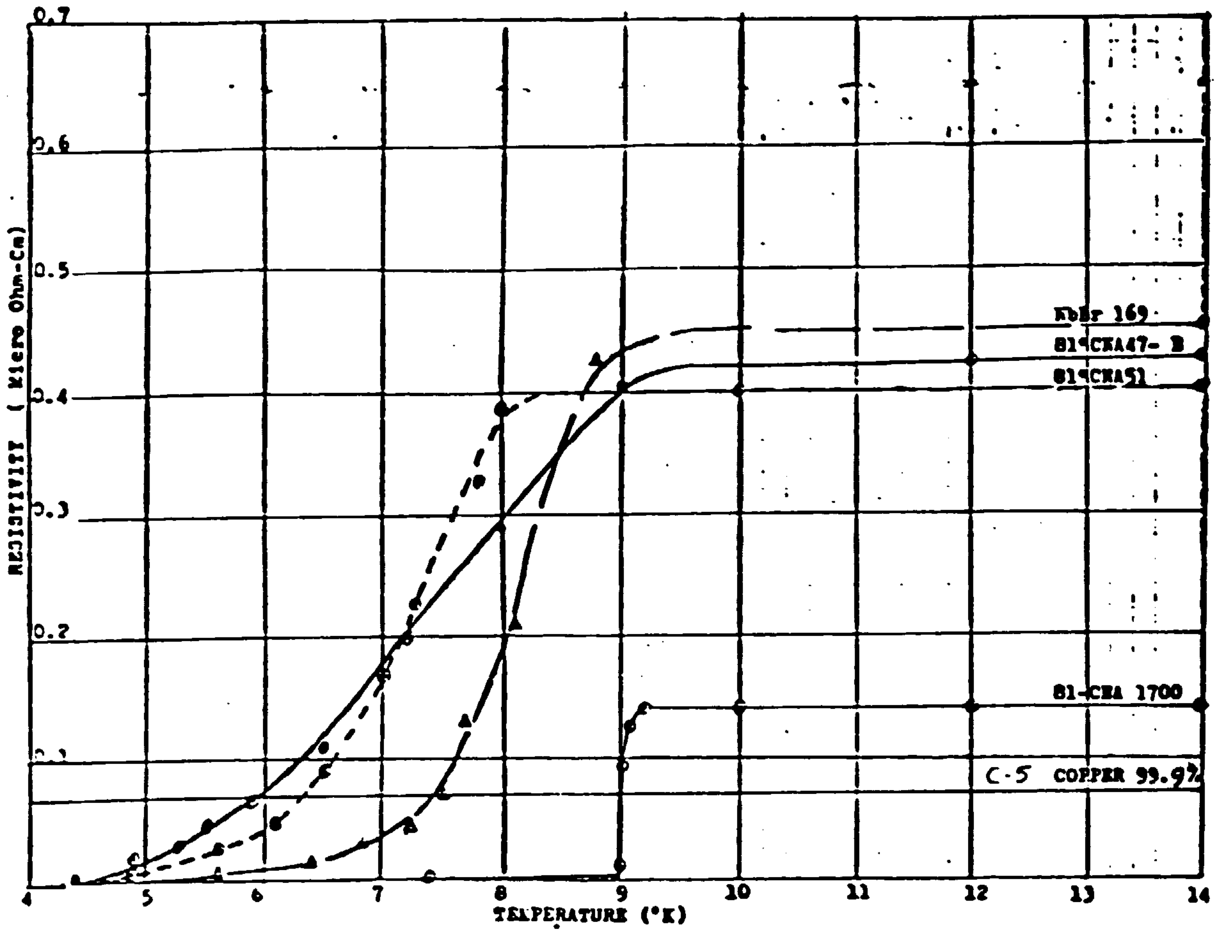


FIGURE 6. Electrical resistivity as a function of temperature for specimens at the following current densities:

Specimen	Current density (A/ cm²)
NbBr 169	7
81CNA47-B	5.3
81CNA51	11.4
81CNA1700	14.7
COPPER 99.9% (c-5)	14.2

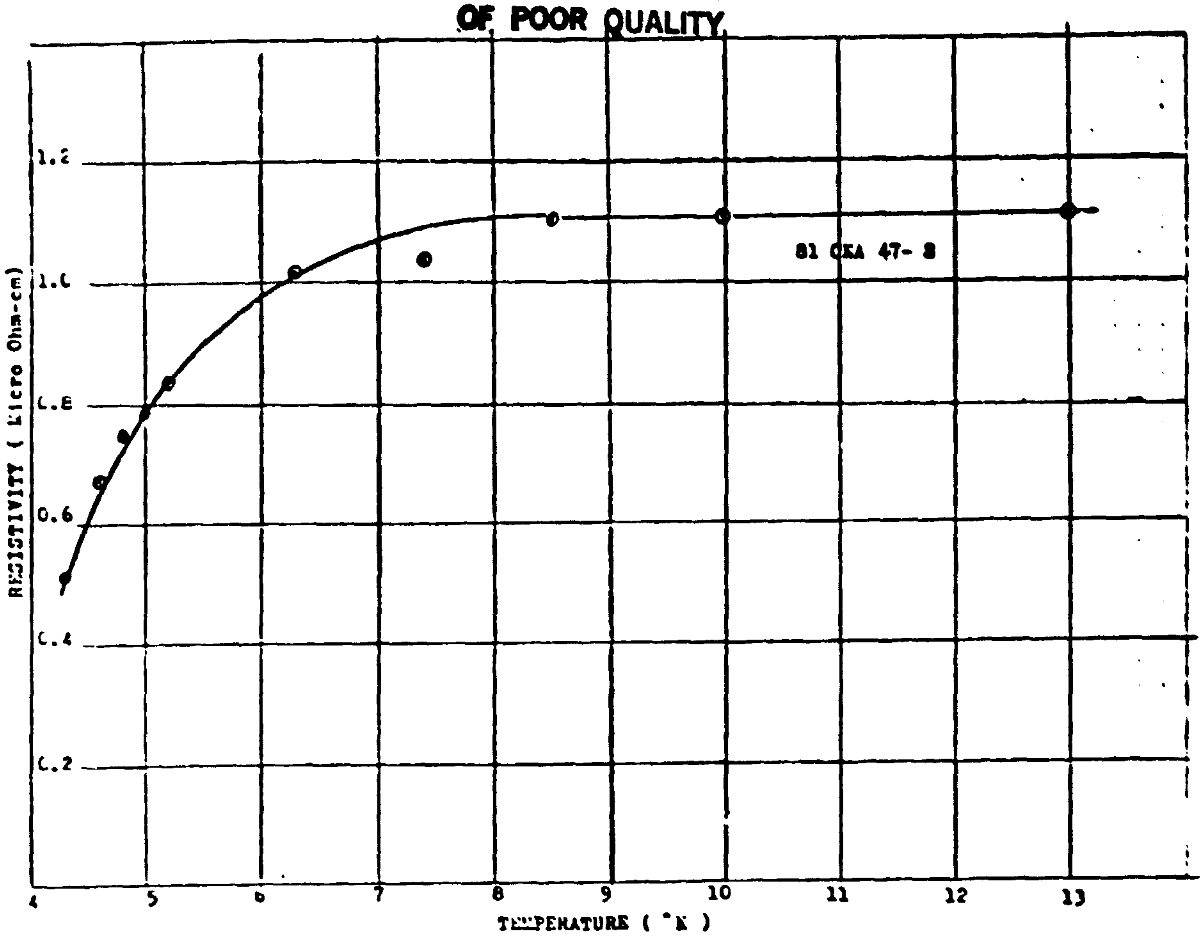


FIGURE 7. Electrical resistivity as a function of temperature for specimen 81CNA47-S at current density of 8.8 A/cm^2.

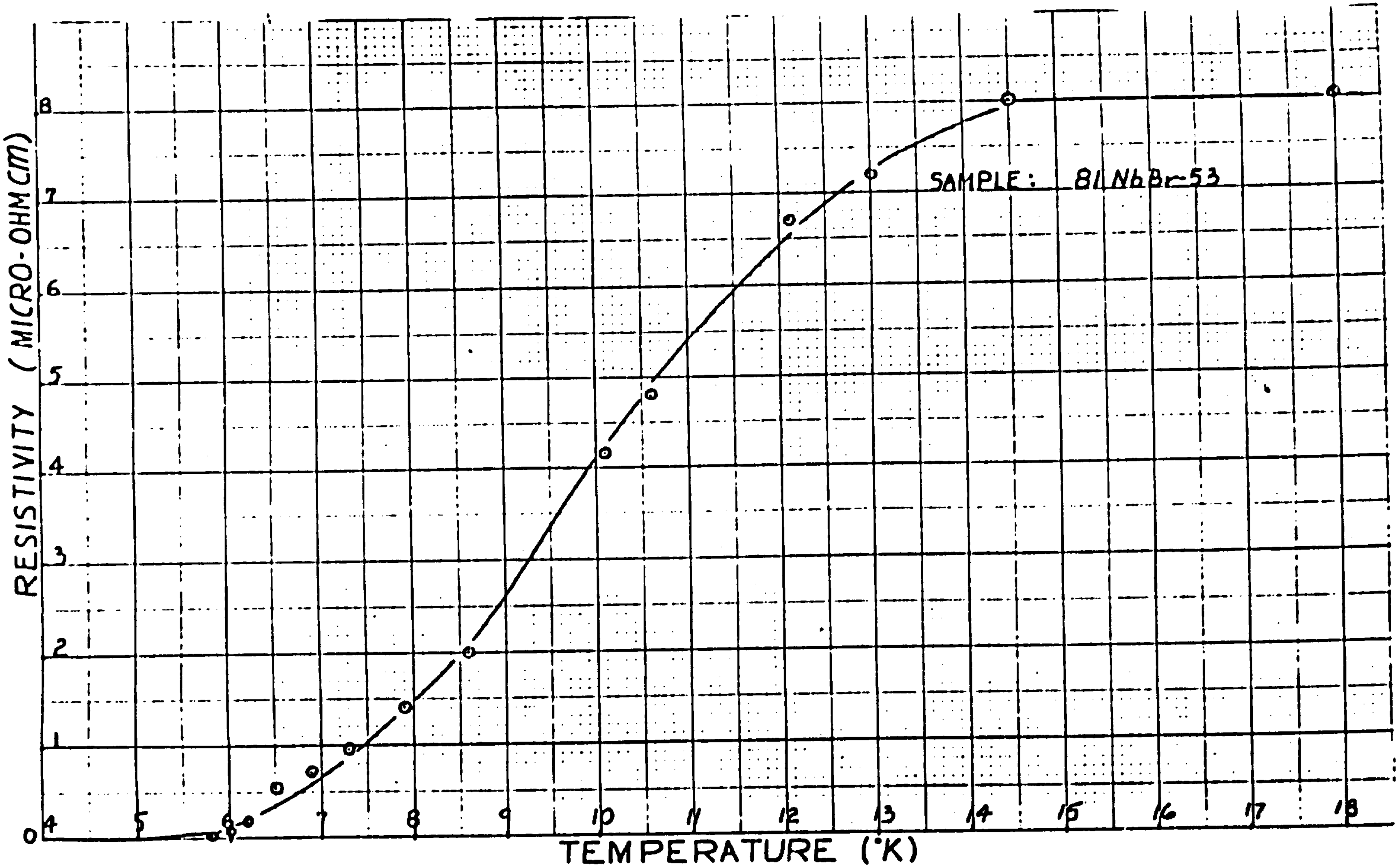


FIGURE 8. Electrical resistivity as a function of temperature for specimen 81NbBr-53 at current density of 2.3 A/cm^2.

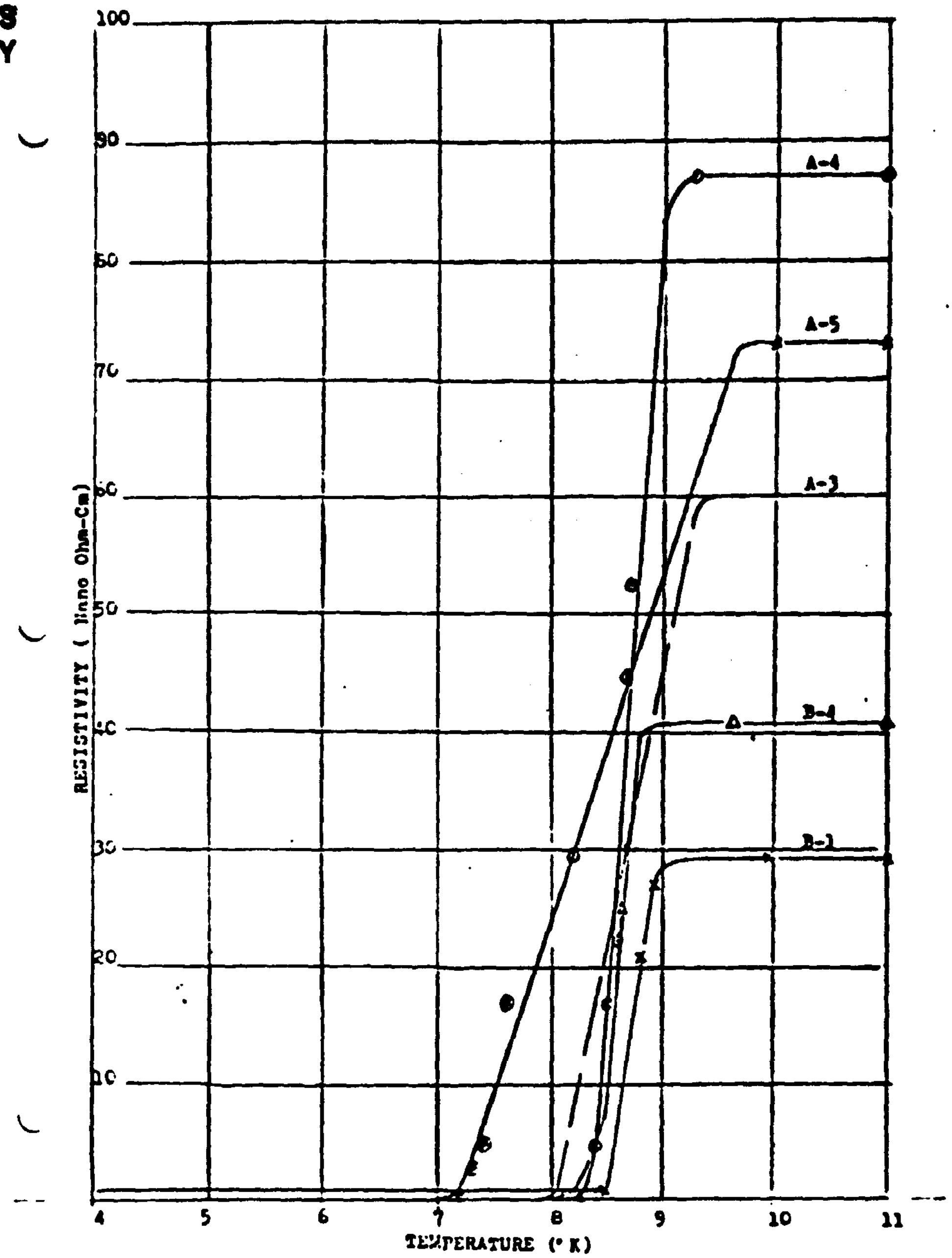


FIGURE 9. Electrical resistivity as a function of temperature for specimens at the following current densities:

Specimen	Current density (A/cm^2)
A-3	62.5
A-4	400
A-5	2604
B-1	8.3
B-4	175

Since the equation is quadratic in S, a six or seven order of magnitude increase in V (into the micro-volt range), although unlikely, is not impossible. T is the absolute temperature and L, the sample length. S_A and S_B are the Seebeck coefficients of the two junctions materials, e.g., the sample and wires (Cu). The apparent resistivity, $\rho_a(T)$, is given as the sum of the true resistivity, $\rho(T)$, and the error (Seebeck) term.

$$\rho_a(T) = \frac{VA}{IL} = \rho(T) + (S_B - S_A)^2 T/K \tag{2}$$

As T approaches T_c, $\rho(T)$ approaches zero and the Seebeck term eventually dominates.

The following improvements (all unsuccessful) were made to identify the "negative resistance" voltage source.

1) Enhancing the interelectrode isolation resistance from 10^4 ohms to about 100 Mohms by replacing the magnet wires (leads) with untwisted magnet wires.
2) Uncoiling the current wire coil near the sample to prevent magneto-resistance.
3) Installing a current reversing switch.
4) Calibrating the nanovoltmeter with a nanovolt source.
5) Increasing the sample depth under L. He to six inches.
6) Replacing the current supply with a wet cell (automobile) for better isolation.
7) Using two continuous synchronized strip chart recorders to monitor sample voltage and current.

Future researchers may wish to use a Faraday cage room, interchange nanovoltmeter, or use a SQUID voltmeter. Routing the two voltage leads

directly to the sample to by pass both the solder joint and the gold coated spring clip (Item 2 in Figure 1) probe should also help. The fact that the negative resistance" showed up in most of the diverse superconducting samples tends to indicate that it is likely apparatus related.

All in all we are 80% confident that the negative voltage is real. We are 30% confident that it is Seebeck related. Our confidence that it is Peltier related is only 10%, however.

IV. CONCLUSIONS

1) A negative response to a certain positive range of current exists for 9 of the 12 specimens tested. Battelle's specimens show a larger current range for this effect. It is not yet known if this is a real effect, therefore, the part of the curves that represent the negative resistance are to be taken as tentative rather than final.

2) NASA's specimens have higher normal resistivity values when transition temperatures are reached. Battelle's specimens have much smaller normal-resistivity value when the temperature (critical) is reached.

3) Critical transition temperature range is large for NASA specimens (vary from 4.3 K to 14.5 K). This could be because of the presence of several phases, each with its own transition temperature. For example, the samples containing tin, show transitions reaching down toward 4°K, the tin transition temperature. In the case of 81NbBr53, the transition stretches upward to 14.5° K, near the transition temperature for the Nb_3Sn compound. Battelle's specimens have smaller critical transition temperature range (vary from 7.2 K minimum to 9.4 K maximum).

4) The sample (NbBr53) which was compacted as niobium and bronze powders and then directionally solidified, showed the highest temperature and the widest transition range. It also had the highest critical current of the directionally solidified samples. This indicates that both stages (preparation of the ingot, and its later processing) determine the final superconducting properties of the material.

5) Of course, the narrow transition of unprocessed 81CNA1700 is expected since the core is initially pure Nb. The later finite resistance and wide transition width of the resulting sample (81CNA51) provides assurance that the Nb was well dissolved in the Cu in the directional solidification process.

V. APPENDIX

This section includes a graph of the resistivity-current characteristics for NASA's specimens which were tested on the previous run (October, 1981). The positive resistivity values are in agreement with the R-I relations reported in this final report. Note that negative resistivity values were not recorded here since the current applied for the specimens to show this effect are small.

The strip charts of current input and voltage response for sample 81CNA47-B at 4.3 K are also shown.

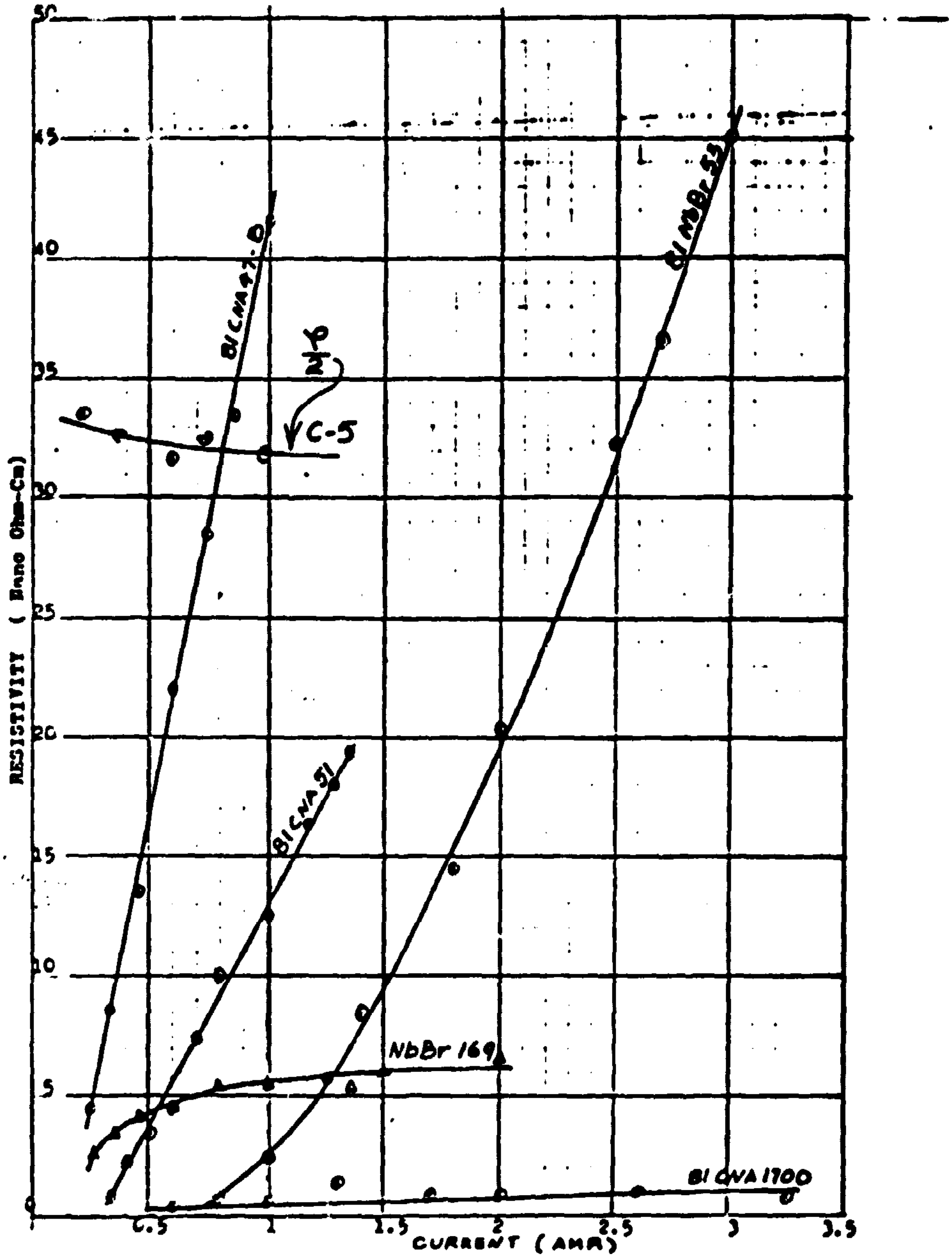
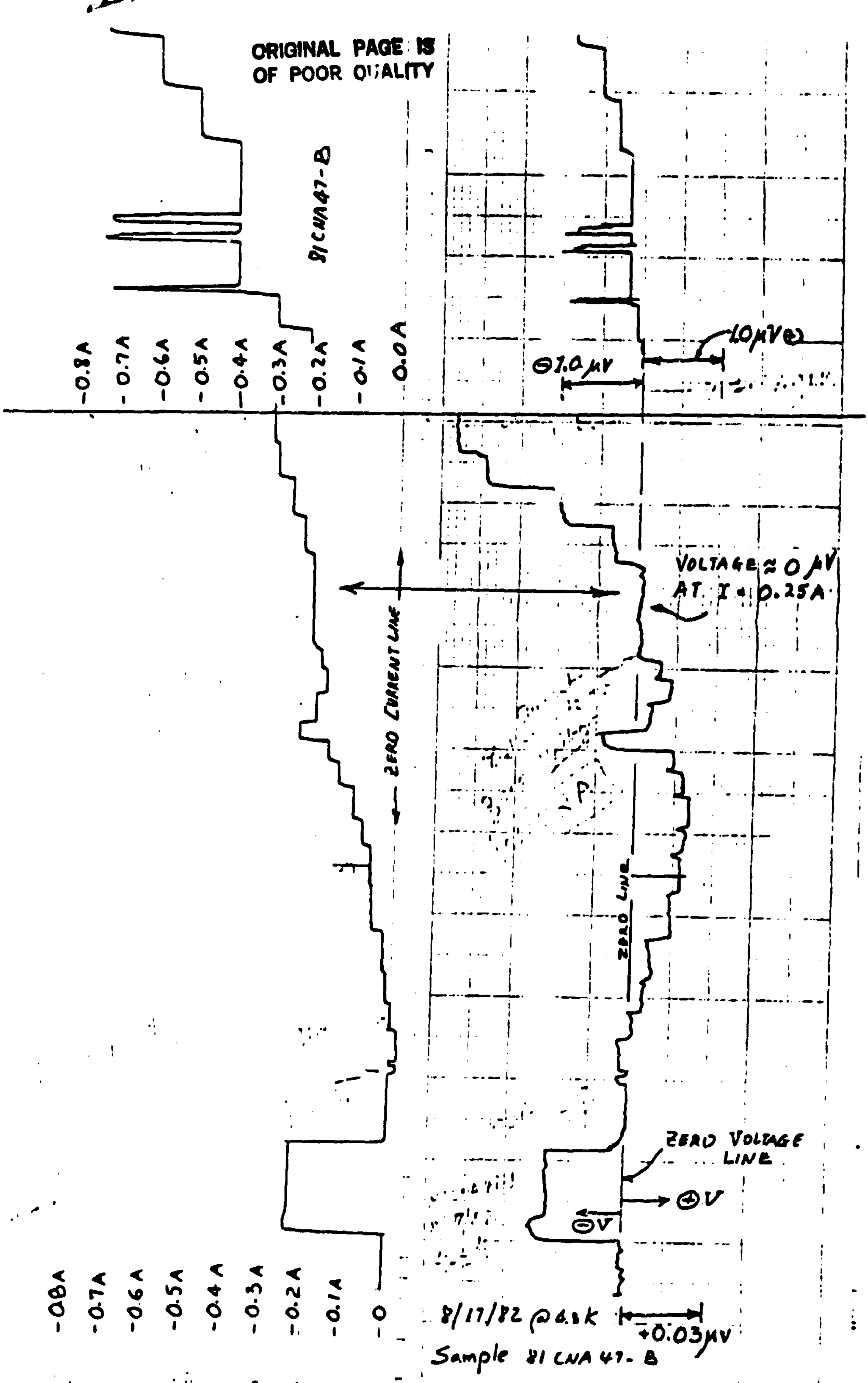
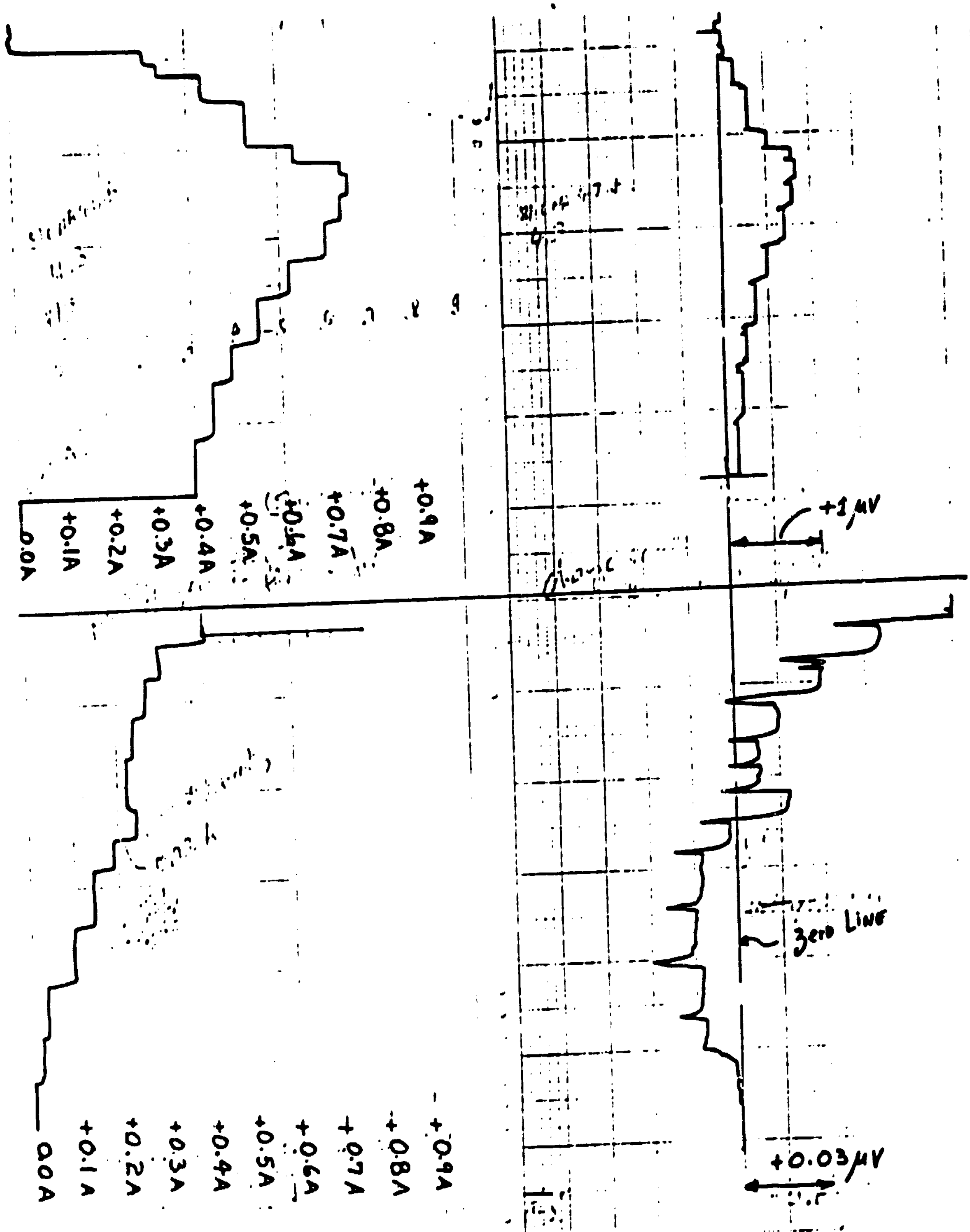


FIGURE 1. Residual resistivity as a function of specimen current at 4.3 K. The current density for each specimen in this graph is found by dividing current to the cross-sectional area of the specimen which is given in table 1. Note that for specimen C-5, the resistivity shown is half the measured value, i.e. $\rho/2$


81CNA47-B
-0.8A
-0.7A
-0.6A
-0.5A
-0.4A
-0.3A
-0.2A
-0.1A
0.0A
10μV
⊖1.0μV
VOLTAGE ≈ 0 μV
AT I ≈ 0.25A
ZERO CURRENT LINE
P
ZERO LINE
ZERO VOLTAGE
LINE
⊕V
⊖V
-0.8A
-0.7A
-0.6A
-0.5A
-0.4A
-0.3A
-0.2A
-0.1A
0
8/17/82 @4.3K
+0.03μV
Sample 81 CNA 47-B

ORIGINAL PAGE IS
OF POOR QUALITY



6/17/82 @ 4.3K

81CNA 47-B

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